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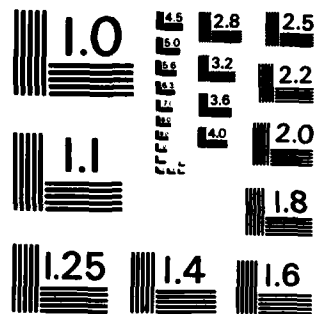
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AIRCRAFT WATER-BASED SOLID FILM LUBRICANTS

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Aircraft and Crew Systems Technology Directorate
NAVAL AIR DEVELOPMENT CENTER
Warminster, PA 18974

10 JUNE 1983

FINAL REPORT
AIRTASK No. A320320A/001B/1F61542000
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Prepared for
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Washington, DC 20361

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) An improved MIL-L-81329 water based extreme temperature range solid film lubricant has been developed. This material provides corrosion protection and improved endurance life for steel on steel components while maintaining the other desirable properties such as oxidation resistance. An upgraded specification covering the properties of this material, which is designated 23C, is provided in appendix A. The goals of this program were twofold;		

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(1) to develop a high temperature water resistant solid film lubricant based on lithium silicate; and (2) to develop a water based non-polluting solid film lubricant to replace MIL-L-8937 for moderate temperature applications. It was found that although lithium silicate films are resistant to solvation by water they are more permeable than sodium silicate films and thus allow corrosion of steel substrates to proceed at a faster rate. The addition of inorganic nitrites as corrosion inhibitors causes an instability of the lithium silicate, with SiO_2 precipitating immediately.

For the moderate temperature range applications of MIL-L-8937, solid film lubricants containing water soluble resins were studied. The best material studied showed an endurance life of only one half that of MIL-L-8937.

The use of an experimental strategy technique was found very beneficial in determining optimum composition ranges with a minimal amount of experimentation.

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SUMMARY

INTRODUCTION

A need exists for a water resistant high temperature solid film lubricant which provides corrosion protection. Specification MIL-L-81329 covers a water based solid film lubricant which contains molybdenum disulfide and graphite and uses sodium silicate as a binder. This material has excellent high temperature capabilities 400° C (752° F), but is seriously deficient in water resistance and corrosion preventive capabilities.

Specification MIL-L-8937 covers an organic solvent/resin bonded solid film lubricant with molybdenum disulfide as the major lubricating pigment. This material was upgraded to include a sulfurous acid/salt spray corrosion test requirement because of corrosion problems associated with solid film lubricant coated fasteners. However, many naval activities are being limited in the amount of organic solvents which can be emitted into the air; therefore, the development of a water based solid film lubricant would alleviate the problem of meeting these requirements.

The dual objectives of this program are to develop a high temperature water based solid film lubricant with good water resistance and corrosion protection properties superior to specification MIL-L-81329 and to develop a water based version of specification MIL-L-8937 material to eliminate the use of organic solvents. This program is being performed under AIRTASK A320320A/001B/1F61542000, Work Unit No. ZM501.

RESULTS

Although lithium silicate films are extremely resistant to solvation by water it was found that their permeability is such that they provide less resistance to corrosion on steel surfaces than sodium silicate films. In an attempt to provide corrosion inhibition utilizing inorganic nitrites, an instability was observed in which SiO_2 (silica) immediately precipitated rendering the silicate useless.

An improved specification MIL-L-81329 type solid film lubricant was developed based on MoS_2 , graphite, potassium nitrite and sodium silicate. This solid film lubricant designated 23C offers corrosion inhibition and improved endurance life for steel on steel friction couples compared to specification MIL-L-81329. A proposed specification has been prepared based on the properties of 23C.

Solid film lubricants using water soluble organic resins as binders were studied as possible replacements for MIL-L-8837 solvent based materials. The best material studied of this type showed only half the endurance life required for specification MIL-L-8937 materials.

RECOMMENDATIONS

It is recommended that this improved solid lubricant designated 23C be used in applications currently requiring specification MIL-L-81329 material. An upgraded specification should be prepared based on the proposed specification found in appendix A.

BACKGROUND

The approach used to increase water resistance of sodium silicate bonded high temperature solid film lubricants centered on the substitution of sodium silicate with lithium silicate. Initial results reported in reference (a) showed that although lithium silicate films provide excellent water resistance compared to sodium silicate films, their corrosion protection properties are degraded. This is due to a high permeability factor associated with these films which was uncovered during this investigation.

An initial investigation on the development of a moderate temperature range water-based solid film lubricant was conducted. This material is a dispersion of solid lubricants in an air-curing, water dilutable, organic resin with an operating temperature range of -70°C (-94°F) to $+250^{\circ}\text{C}$ (482°F). In the preparation of steel specimens for endurance life testing, it was noted that the specimens started to corrode when the solid film was applied. Careful preparation of specimens was required to eliminate this initial action of the solid film lubricant on the test specimens. Falex endurance testing of this formulation resulted in an average wear life of 120 minutes. Specification MIL-L-8937 films are required to run at least 250 minutes.

EXPERIMENTAL

1. The Falex endurance life tests were performed in accordance with ASTM D2625 procedure A. The steel specimens were grit blasted with 120 steel grit to produce a surface finish of 50 to 60 RMS prior to solid lubricant deposition.
2. The Falex load-carrying capacity tests were performed in accordance with ASTM D2625 procedure B. The steel specimens were pretreated as described above.
3. The sulfurous acid-salt spray tests were performed in accordance with Test Method 5331 of Federal Test Method Standard 791. AISI 1010 steel disks were used as substrates. One cycle consists of a two hour exposure to the sulfurous acid-salt spray and a minimum two hour drying time.
4. MOS_2 conformed to MIL-M-7866.
5. Graphite conformed to SS-G-659.
6. Potassium nitrite conformed to certified ACS grade.
7. Graphite-fluoride was a white powder which was obtained from Alpha Products Company, Inc. with a fluorine to carbon ratio of greater than one.
8. Lithium silicate solutions were obtained from the Lithium Corporation of America (LITHCOA).
9. Sodium silicate was obtained from Philadelphia Quartz Company.

RESULTS AND DISCUSSION

HIGH TEMPERATURE RANGE WATER-BASED SOLID FILM LUBRICANT

Corrosion Inhibition of Lithium Silicate Films

Table I lists the composition and properties of various alkali metal silicate solutions investigated in this program. In an effort to impart corrosion protection properties to lithium silicate films various water soluble inorganic salts such as potassium nitrite (KNO_2), sodium phosphate Na_3PO_4 , and sodium molybdate Na_2MoO_4 which are known corrosion inhibitors, were added individually to silicate solutions. As shown in figure 1 a white precipitate is formed when potassium nitrite is added to a solution of lithium silicate A. The addition of salts to lithium silicate solutions produce an instability in which silica (SiO_2) is immediately precipitated from solution and the properties of lithium silicate solutions are lost. With sodium silicate solutions the addition of potassium nitrite does not affect the stability of SiO_2 in solution.

Because of the inability to provide corrosion inhibition for lithium silicate films, the direction of this program changed toward upgrading MIL-L-81329 sodium silicate bonded films.

Graphite Fluoride Films

The first approach toward providing a corrosion inhibiting MIL-L-81329 film involved the use of graphite-fluoride as a substitute for "normal" graphite in a typical MIL-L-81329 formulation. Graphite-fluoride is a covalent intercalation compound in which the conductivity of graphite has been drastically reduced. It was found that the lubricating solids would not disperse in water-based formulations containing graphite fluoride. The addition of a surfactant was used to remedy this situation. The Falex endurance life for this formulation compared to a MIL-L-81329 type of solid film lubricant is shown in table II. A 42 percent reduction in endurance life can be observed on substituting graphite with graphite-fluoride. An 80 percent reduction in endurance life is found for a phenolic resin bonded film comparing only graphite to graphite-fluoride. Efforts to improve corrosion protection properties using graphite fluoride stopped when the reduced endurance life was observed.

Improved Sodium Silicate Bonded Solid Film Lubricant

An experimental strategy was introduced in this development program (reference (a) which was found extremely beneficial in defining composition ranges for obtaining an optimum endurance life with solid film lubricant coating systems. Using a three component approach, ie., MoS_2 , graphite and silicate binder, various formulations were prepared based on keeping the sum of the weight of components constant. A composition of 60 percent MoS_2 , 10 percent graphite and 30 percent sodium silicate exhibited the highest endurance life (68 minutes). A further refinement of this procedure which involved narrowing the composition limits in the above concentration range resulted in an optimum endurance life of 75 minutes for 53.3 percent MoS_2 , 18.4 percent graphite, and 28.3 percent sodium silicate (See table III). This composition which is designated 23B represents a 3 fold improvement in endurance life for steel specimens compared to the original MIL-L-81329 composition 23A (55.0 percent MoS_2 , 5.5 percent graphite, and 39.5 percent sodium silicate).

Corrosion Inhibiting Sodium Silicate Bonded Solid Film Lubricant

The improved formulation designated 23B, was used to study the corrosion inhibition properties of inorganic nitrites of sodium and potassium. Initially, sodium nitrite was investigated

TABLE I. COMPOSITION AND PROPERTIES OF ALKALI METAL SILICATES

	<u>Lithium Silicate A</u>	<u>Lithium Silicate B</u>	<u>Lithium/ Sodium Silicate</u>	<u>Sodium Silicate (MIL-L-81329)</u>
Li ₂ O, %	2.2	1.6	1.8	---
Na ₂ O, %	---	---	1.2	10.9
SiO ₂ , %	20.7	18.8	19.6	31.9
Total Solids %	22.9	20.4	22.6	42.8
Molar Ratio:				
SiO ₂ /Li ₂ O	4.6:1	5.9:1	---	---
SiO ₂ /Na ₂ O	---	---	---	3.0:1
SiO ₂ /Li ₂ O/Na ₂ O	---	---	5.4:1:0.3	---
pH	10.7	10.7	10.7	11.5
Density, 25°C (77°F) g/cc	1.17	1.18	1.19	1.49
Viscosity, 30°C (86°F) cps	22.5	3.5	4.6	4.8

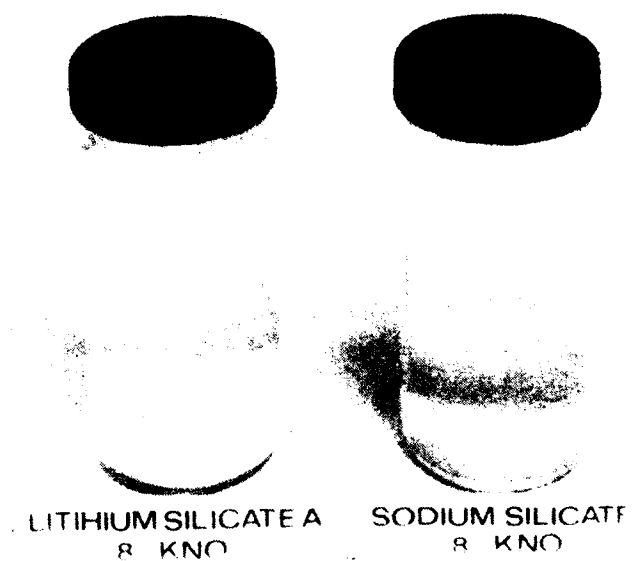


Figure 1. Comparison of the Appearance of Lithium Silicate and Sodium Silicate Solutions on the Addition of KNO_2

TABLE II. FALEX TEST RESULTS ON GRAPHITE
AND GRAPHITE-FLUORIDE SOLID FILM LUBRICANTS

Load: 4488N (1000 lb) Gage Load
 Speed: 290 RPM
 Temperature: Ambient
 Pins: AISI 3135
 V-Blocks: AISI 1137
 Surface Pretreatment: Grit Blasted, 120 Steel Grit

<u>MoS₂</u> <u>g</u>	<u>Graphite</u> <u>g</u>	<u>Graphite-Fluoride</u> <u>g</u>	<u>Sodium Silicate</u> <u>Binder g</u>	<u>Phenolic</u> <u>Binder</u> <u>g</u>	<u>Endurance Life</u> <u>Minutes</u>
70.0	7.0	—	50	—	24 ± 6
70.0	—	7.0	50	—	14 ± 6
—	14.2	—	—	67.0	51 ± 8
—	—	14.2	—	67.0	10 ± 4

TABLE III. COMPOSITIONS OF SODIUM SILICATE BONDED
SOLID FILM LUBRICANTS AND FALEX
ENDURANCE LIFE

Composition No.	MoS ₂ %	Graphite %	Sodium Silicate %	Falex Endurance		
				Life, Min.		
				Run 1	Run 2	AVG
1	70	5	25	39	27	33
2	50	25	25	5	8	7
3	50	5	45	13	13	13
4	60	15	25	77	69	73
5	60	5	35	13	16	15
6	50	15	35	60	65	63
7	56.7	11.6	31.7	57	55	56
8	63.3	8.4	28.3	49	47	48
9	53.3	18.4	28.3	82	67	75
10	53.3	8.4	38.3	42	30	36

Composition Limits

MoS₂ = 50 to 70%

Graphite = 5 to 25%

Sodium Silicate = 25 to 45%

but because of its lower melting point, 271°C (570°F), potassium nitrite with a melting point of 440°C (824°F) was substituted. As shown in a previous section of this report, the addition of nitrite to the sodium silicate solution does not cause any instability. A formulation designated 23C based on 8 percent KNO₂ (of solids) was found to provide the desired corrosion protection. Figure 2 shows the results of sulfurous acid-salt spray corrosion tests on the formulations. After only two cycles, the steel disk with the non-inhibiting coating is severely corroded while the disk with the inhibited coating shows no sign of corrosion after six cycles. An improvement is also noted for disks which have score marks on them. Having demonstrated the corrosion protection properties of 23C, it was necessary to ascertain whether the addition of potassium nitrite (KNO₂) would be detrimental to the endurance life of the film. As shown in table IV, the endurance life of the corrosion inhibited film is essentially the same as that of the non-inhibited film. Also, the load-carrying capacity of 23C is higher than 23B by 4488N (1000 pounds).

Moderate Temperature Range Water-Based Solid Film Lubricant

Various acrylic, polyurethane and alkyd based water soluble binders were studied as substitutes for organic resin bonded solid lubricants. The goal was to provide a water-based solid film lubricant with properties equivalent to MIL-L-8937 material. Only one material was found to have substantial endurance life and this was about half of the minimum required by MIL-L-8937.

REFERENCES

- (a) Conte, Alfeo, A., Jr., "Water-Based Solid Film Lubricants," Report No. NADC-81300-60 of 27 April 1982.

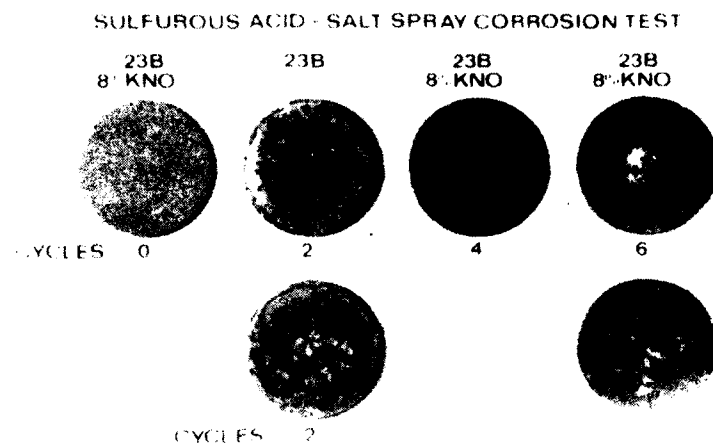


Figure 2. Sulfurous-Acid Salt Spray Corrosion Tests on 23B and 23C
(23B + 8% KNO₂) Solid Film Lubricants

TABLE IV. FALEX TEST RESULTS ON CORROSION
INHIBITED HIGH TEMPERATURE SODIUM
SILICATE BONDED SOLID FILM LUBRICANT

Speed: 290 RPM
 Temperature: Ambient
 Pins: AISI 3135
 V-Blocks: AISI 1137
 Surface Pretreatment: Grit Blasted, 120 Steel Grit

Designation	MoS ₂ g	Graphite g	Sodium Silicate g	KNO ₂ g	Endurance Life, Minutes 4488N (1000 lb)	Load Carrying Capacity N (lb)
23B	53.3	18.4	28.3	—	75 ± 11	13,464 (3000)
23C	53.3	18.4	28.3	7.3	71 ± 5	17,952 (4000)

APPENDIX A

PROPOSED MILITARY SPECIFICATION

LUBRICANT, AIRCRAFT, SOLID FILM, EXTREME ENVIRONMENT,
CORROSION INHIBITING

1.0 SCOPE

1.1 Scope. This specification covers an aircraft corrosion resistant solid film lubricant for use in extreme aircraft environments at temperatures ranging from -185C (-301F) to 400C (752F).

2.0 APPLICABLE DOCUMENTS

2.1 Issues of Documents. The following documents, of the issue in effect on the date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein:

SPECIFICATIONS

FEDERAL

- QQ-A-250/5 — Aluminum Alloy Alclad 2024, Plate and Sheet
- PPP-C-96 — Cans, Metal, 28 Gage and Lighter

MILITARY

- MIL-M-3070 — Mixer, Liquid, Revolving-Shaft and Agitator Types
- MIL-S-5059 — Steel, Corrosion-Resistant (18-8), Plate, Sheet and Strip (Asq.)
- MIL-A-8625 — Anodic Coatings, for Aluminum and Aluminum Alloys
- MIL-T-81533 — Trichloroethane -1,1,1 (Methy Chloroform) Inhibited, Vapor Degreasing

STANDARDS

FEDERAL

- Fed. Test Method — Lubricants, Liquid Fuels, and Related Products;
- Std. No. 791 — Methods of Testing

MILITARY

- MIL-STD-105 — Sampling Procedures and Tables for Inspection by Attributes
- MIL-STD-290 — Packaging, Packing, and Marking of Petroleum and Related Products

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer).

2.2 Other Publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS

- ASTM D270 — Petroleum and Petroleum Products, Sampling
- ASTM D2510 — Adhesion of Dry Solid Film Lubricants, Test for
- ASTM D2511 — Thermal Shock Sensitivity of Dry Solid Film Lubricants, Test for
- ASTM D2512 — Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold Technique), Test for
- ASTM D2625 — Endurance (Wear) Life and Load-Carrying Capacity of Dry Solid Film Lubricants (Falex Test), Determining

(Copies of ASTM publications may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103).

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

3. REQUIREMENTS

3.1 First Article. When specified (see 6.2), a first article sample shall be tested by a government laboratory (see 4.2). The contractor shall furnish sample units for first article inspection and approval (see 4.2 and 6.2).

3.2 Materials. The lubricant shall consist essentially of lubricating solids and corrosion inhibitor in a suitable binder, at spraying consistency. Organic materials are not suitable for lubricant compositions furnished under this specification. The applied film shall be capable of being cured under conditions of the following heating schedule:

- a. Air dry for 1/2 hour @ $25 \pm 3^{\circ}\text{C}$ ($77 \pm 5^{\circ}\text{F}$)
- b. 80°C (176°F) for 2 hours followed by
- c. 150°C (302°) for 2 hours

3.2.1 Preparation. Appendix A covers a suitable formulation for preparation of bonded solid film lubricant (see 6.4).

3.3 Appearance. When examined in accordance with 4.4.2, the bonded solid film lubricant, applied as specified in 4.4.1, (see 6.5) shall be uniform in color, smoothness, and thickness, and free

of cracks, scratches, pinholes, blisters, bubbles, runs, sags, foreign matter, grit, rough particles, separation of ingredients, or any other surface imperfections. Unless otherwise specified, the thickness of the finished film shall be between 0.0015 to 0.0020 inch.

3.4 Flammability. When tested as specified in 4.4.3, the lubricant shall be nonflammable.

3.5 Film Adhesion. When tested in accordance with 4.4.4, the bonded film lubricant shall not be lifted from the test panel by the tape employed. A uniform deposit of powdery material may cling to the tape, but the lifting of any flakes or particles of the lubricant, which exposes any anodized aluminum substrate on the test panel shall indicate unsatisfactory adhesion.

3.6 Thermal Stability. When tested between minus -185°C (-301°F) and 400°C (752°F) in accordance with 4.4.5, the bonded solid film lubricant shall not flake, crack, or soften and shall show satisfactory adhesion as measured by 3.5.

3.7 Endurance Life. When tested in accordance with 4.4.6 in the Falex Lubricant Tester, the bonded solid film lubricant shall have an average endurance life of 60 minutes at 1,000 pounds.

3.8 Load-Carrying Capacity. When tested in accordance with 4.4.6, the bonded solid film lubricant shall have a load-carrying capacity of at least 3,000 pounds gage load.

3.9 Sulfurous Acid - Salt Spray. When steel specimens coated with dry film lubricant are exposed to sulfurous acid - salt spray for 4 cycles in accordance with 4.4.7. There shall be no resultant pitting or visible corrosion.

3.10 Shock Sensitivity with Liquid Oxygen. When tested as specified in 4.4.8, the bonded solid film lubricant shall show no adverse reactions to 20 test drops at 70 foot-pounds.

3.11 Storage Stability. The lubricant when stored for 12 months in accordance with 4.4.9 in solid film solution form at $25 \pm 2^{\circ}\text{C}$ ($77 \pm 3^{\circ}\text{F}$) in a full, closed container and then subjected to mechanical shaking shall remain a homogeneous blend and shall show no evidence of gelatin. The stored lubricant, after application as specified in 4.4.1, shall form a bonded solid film lubricant which will meet the film adhesion (3.5), thermal stability (3.6), and endurance life (3.7) requirements of this specification.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Classification of Inspection. The examination and testing shall be classified as follows:

- a. First article inspection (4.2.1)
- b. Quality conformance inspection (4.2.2)

4.2.1 First Article Inspection. The first article inspection shall consist of testing the first article sample for Section 3 requirements deemed necessary by the testing activity designated (see 4.3.1 and 6.2) and certification by the manufacturer of either alternative a. or b., as follows:

- a. The manufacturer shall certify that his formulation is in accordance with Appendix A. Under these conditions the Government agrees that the lubricant meets the requirements of storage stability (3.11). A certified copy of a test report showing conformance to all other requirements of this specification shall be supplied.
- b. If the manufacturer uses some other formulation and means of application, he must supply a certified test report that the lubricant has successfully completed tests indicating that it meets all the requirements of this specification. Delivery of the lubricant will be deferred pending completion of the first article inspection. If the results of the first article inspection are in accordance with the requirements of this specification, the consistency of production quality will be thereby verified. Failure of the lubricant to pass a first article inspection shall require that the acceptance and further shipment be withheld until the contractor has corrected the condition which led to the failure.

4.2.2 Quality Conformance Inspection. The quality conformance inspection (lot-by-lot) shall include examining and testing the quality conformance samples (4.3.2.3) for conformance to all the Section 3 requirements, except stability (3.11), and an examination of the samples of filled containers (4.3.2.4) for conformance to Section 5.

4.3 Sampling and Acceptability Criteria

4.3.1 First Article Samples. First article samples shall consist of three quarts selected at random from the first lot of lubricant processed under a contract or order. The sample shall be accompanied by either a copy of certification to the formulation (see alternate a. of 4.2.1) or a copy of a certified test report (see alternate b. of 4.2.1). The first article inspection samples, reports and certifications shall be forwarded to the Naval Air Development Center, Code 60612, Warminster, Pennsylvania 18974. The samples shall be plainly marked by securely attached durable tags or labels marked with the following information:

Sample for first article
LUBRICANT, SOLID FILM, EXTREME ENVIRONMENT, CORROSION
INHIBITING
MIL-L-XXXXXXX
Name of Manufacturee
Product Code Number
Date of Manufacture
Contract or Order Number
Batch Number

4.3.2 Quality Conformance Samples. The quality conformance samples shall consist of a sample for tests (4.3.2.3) and a sample for examination of filled containers (4.3.2.4). Samples shall be labeled completely with information identifying the purpose of the sample, name of manufacturer, name of product, specification number, lot and batch number, date of sampling, and contract number. Individual samples shall not be mixed, but shall be placed in separate air-tight and water-tight containers, which shall be nearly filled, then covered and sealed to prevent atmospheric effects.

4.3.2.1 Inspection Lot. A lot shall consist of the material produced by one manufacturer under essentially the same manufacturing conditions. Each batch shall constitute a lot.

4.3.2.2 Bulk Lot. An indefinite quantity of a homogeneous mixture of material offered for acceptance in a single isolated container; or manufactured in a single plant run (not exceeding 24 hours) through the same processing equipment, with no change in ingredient material, is a "bulk lot".

4.3.2.3 Sample for Tests. The sample for tests shall consist of one can of lubricant taken at random from each lot of packaged lubricant or, if obtained from a bulk tank, in accordance with ASTM-D270.

4.3.2.4 Sample for Examination of Filled Containers. A random sample of filled containers and a sample of shipping containers fully prepared for delivery shall be selected from each lot of lubricant in accordance with MIL-STD-105 at inspection Level I and acceptable quality level (AQL) of 2.5 percent defective.

4.4 Test Methods. Unless otherwise specified, all examinations and tests shall be performed at a temperature of $25 \pm 3^{\circ}\text{C}$ ($77 \pm 5^{\circ}\text{F}$). The physical and chemical values specified in Section 3 apply to the average of the determinations made on the samples.

4.4.1 Preparation of Test Panels. Samples of the lubricant in sprayable form shall be used to prepare bonded film lubricant specimens on test panels in accordance with the following application procedure:

- a. The panels shall be made from:
 - (1) Aluminum alloy conforming to QQ-A-250/5 and anodized to conform to MIL-A-8625, Type I measuring approximately 0.020 by 3 by 6 inches.
 - (2) Corrosion resistant steel conforming to MIL-S-5059, composition 321, condition annealed, finish No. 2 dull, measuring approximately 0.036 by 3 by 6 inches.
- b. The panels shall be precleaned using trichloroethane conforming to MIL-T-81533. The stainless steel panels shall be sandblasted with 120 steel grit. The surface of the panel shall be held at a distance of 2.5 inches from the discharge nozzle (orifice opening, of 1/4 inch diameter) delivering grit at an air pressure of 80-90 psi. After the panels have been sandblasted the panels shall be cleaned with a trichloroethane rinse and dried with a stream of dry air that is free of oil. A spray application technique shall be used to coat the panels for the tests of this specification. Two or three coats may be required to reach the desired dry film thickness of 0.0015 to 0.0020 inch (see 6.4). Air drying, at a temperature of $25 \pm 3^{\circ}\text{C}$ ($77 \pm 5^{\circ}\text{F}$) for ten minutes, shall be allowed between coats. After the final coat has been applied, the coated panels shall be allowed to air dry for 30-35 minutes. The coated panels shall then be placed in an oven at 80°C (176°F) for 120-125 minutes in an oven at 158°C (302°F). The coated panels shall be removed from the oven and allowed to cool to room temperature. At least two coated panels shall be used in each test method requiring test panel specimens.

4.4.2 Film Appearance and Thickness. The bonded solid film lubricant specimens shall be examined visually and microscopically at a magnification of 12X for uniformity in color, smoothness, and thickness, and evidence of cracks, scratches, pinholes, blisters, bubbles, runs, sags, foreign matter, grit, rough particles, separation of ingredients and any other surface imperfections. Film thickness shall be determined by a magnaguage or by the difference in thickness as measured between an unlubricated and anodized aluminum panel and the same panel coated with the dry film lubricant. To insure measurements in the same portion of the panel, a template shall be made the same size as the panel with four 3/8-inch holes centered 1-1/2 inches in from each 3-inch end and 5/8 inch in from each 6-inch end. Using a precision micrometer with a 0.0001 inch graduation, the thickness shall be measured at each opening in the template with the template in position on the uncoated panel. After the dry film has been applied to the panel and cured, the thickness shall be remeasured at the same positions. The difference in the readings represents the film thickness of the lubricant.

4.4.3 Flammability. A 3 to 5 gram specimen of the lubricant sample shall be placed on a 60 to 65 Taylor mesh stainless steel screen. The area covered by the specimen shall be approximately 1-1/2 inches in diameter. The underside of the screen shall be cautiously heated with a Bunsen burner in a manner which will not cause the flame to project through or above the screen. As soon as the compound begins to decompose or volatilize, a second burner shall be used to ignite any vapors evolved. The specimen will be considered nonflammable if the burning ceases as both burners are withdrawn from the screen. Heating shall be continued until the sample is at a red heat; both upper and lower surfaces shall be examined for a self-supporting flame.

4.4.4 Film Adhesion. Four anodized aluminum panels to which the solid film lubricant has been bonded (see 4.4.1) shall be adhesion tested in accordance with ASTM Method D2510.

4.4.5 Thermal Stability. Thermal stability shall be performed in accordance with ASTM Method 2511.

4.4.6 Endurance Life and Load-Carrying Capacity. The bonded solid film lubricant shall be tested for endurance life and load-carrying capacity in accordance with ASTM D2625 except that the cure schedule shall be in accordance with Appendix A. The V-blocks may be either phosphated or grit blasted with 120 steel grit to produce a surface roughness of 50-60 RMS.

4.4.7 Sulfurous Acid — Salt Spray Corrosion Test — The sulfurous acid — salt spray test on steel shall be determined in accordance with test method 5331 of Federal Test Method Standard 791.

4.4.8 Shock Sensitivity with Liquid Oxygen. The bonded film shall be tested as specified in ASTM Proposed Method D2512 for compatibility of materials with liquid oxygen.

4.4.9 Storage Stability. A full, closed 1-quart container of the solid film solution shall be stored undisturbed at a temperature of $25 \pm 2^\circ\text{C}$ ($77 \pm 5^\circ\text{F}$) for a period of 13 months. The container of lubricant shall be subjected to mechanical agitation using an apparatus similar to MIL-C-3070, Type II, size as required, or equal, for 5 minutes. The container shall then be opened and the lubricant examined for homogeneity. The lubricant shall then be applied as specified in 4.4.1. The bonded lubricant film shall then be tested for film adhesion (4.4.4), thermal stability (4.4.5), and endurance life (4.4.6). Observations shall be made of homogeneity of solution as well as for evidence of gelatin.

5. PACKAGING

5.1 Packaging. Unless otherwise specified (see 6.2), the lubricant shall be packaged, packed, and marked in accordance with MIL-STD-290. The lubricant shall be packaged in 1-pint polyethylene bottles or in Type 1X, Class 3 can, conforming to PPP-C-96.

5.1.1 In addition to the marking specified in 5.1, the individual containers shall be marked to show "Date of Manufacture" and the marking shall include the following information:

USE THIS LUBRICANT WITHIN 12 MONTHS FROM THE DATE OF MANUFACTURE

CURE SCHEDULE

- a. Air dry for 1/2 hour at $25 \pm 3^{\circ}\text{C}$ ($77 \pm 5^{\circ}\text{F}$)
- b. 80°C (76°F) for 2 hours followed by
- c. 150°C (302°F) for 2 hours.

6. NOTES

6.1 Intended Use. The lubricant covered by this specification is intended for use in aircraft, liquid oxygen systems, space vehicles, bearing assemblies and other equipment where the environments of temperature, nuclear radiation, and vacuum preclude the use of conventional lubricants and organic solid film lubricant. It is intended to reduce wear and prevent galling and seizing of metal surfaces. Do not use the lubricant on materials that may be adversely affected by exposure to the specific cure temperature of 140°C (300°F). It should not be used with oils or greases unless field use indicates otherwise.

6.2 Ordering Data. Procurement documents should specify the following:

- a. Title, number and date of this specification.
- b. Quantity desired.
- c. Where preproduction inspection (first article) samples shall be forwarded (see 4.3.1).
- d. Size and type of container in which lubricant is to be furnished (see 5.1).
- e. Packaging, packing, and marking requirements, if other than as specified in Section 5.
- f. Items of data required (see 6.3).

6.2.1 Contract Provision. Contracts shall specify the following provision for first article inspection.

6.2.1.1 First Article. When a first article is required for inspection and approval (see 3.1, 4.2, 6.2 and 6.3), the contract shall specify the following provisions for first article inspection.

When a contractor is in continuous production of the compound from contract to contract, consideration should be given to waive the first article inspections. If inspection is required, indicate:

- (a) If first article inspections are conducted at the contractor's plant or a government approved laboratory, an inspection report shall be forwarded to the procuring activity for verification.
- (b) That the approval of first article samples or the waiving of the first article inspection shall not relieve the contractor of his obligation to fulfill all other requirements of the specification and contract.

6.2.2 The lubricant should not be ordered for use beyond 12 months from the date of manufacture.

6.3 Contract Data Requirements. When this specification is used in a procurement which incorporates a DD Form 1423 and invokes the provisions of 7-104.9(n) of the Armed Services Procurement Regulations, the data requirements identified below will be developed as specified by an approved Data Item Description (DD Form 1664) and delivered in accordance with the approved Contract Data Requirements List (DD Form 1423) incorporated into the contract. When the provisions of ASPR-7-104.9(n) are not invoked, the data specified below will be delivered by the contractor in accordance with the contract requirements. Deliverable data required by this specification is cited in the following paragraphs:

Paragraph	Data Requirement	Applicable DID
4.2.1	First Article Inspection Reports	DI-T-5329 - Inspection Test Reports

DD Form 1423 should state in block 16 "delete the word equipment".

(Copies of data item descriptions required by the contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer).

6.4 The formulation and application of the lubricant under this specification shall be optional with the manufacturer, but shall be restricted by the requirements of this specification. However, a formulation and application instructions which has proven suitable are described in Appendix A. Declaration by the contractor that the lubricant has been processed in accordance with Appendix A will facilitate first article inspection.

6.5 Spray application (two coats) produces a film thickness in the range of 0.0015 - 0.0020 inch. This thickness may be reduced by burnishing the cured film. A suitable burnishing technique is as follows:

- a. Vapor degrease "Grade 0" steel wool.
- b. Using clean, white, lint-free cotton gloves, rub the solid film lubricant surface gently and uniformly with the steel wool. A micrometer or other suitable measuring technique shall be used to determine film thickness.
- c. Remove loose film particles and steel wool by jet of air which is oil and water-free.

- d. Continue the burnishing technique until the desired film thickness is achieved.

6.6 International Standardization Agreement. Certain provisions (1.1) of this specification are the subject of international standardization agreement Stanag 1135. When amendment, revision, or cancellation of this specification is proposed which will affect or violate the international agreement concerned, the preparing activity will take appropriate reconciliation action through international standardization channels, including departmental standardization offices, if required.

APPENDIX A1

FORMULATION, PREPARATION, AND APPLICATION OF CORROSION INHIBITED MoS₂
GRAPHITE-SODIUM SILICATE SOLID FILM LUBRICANT (SPRAY CONSISTENCY)

10. SCOPE

10.1 This appendix covers the formulation and preparation of a corrosion inhibited molybdenum disulfide-graphite-sodium silicate bonded solid film lubricant that has proved suitable for meeting the requirements of the basic specification.

20. APPLICABLE DOCUMENTS

20.1 The following specifications, of the issue in effect on date of invitation for bids or request for proposal, form a part of this appendix to the extent specified herein.

SPECIFICATIONS

FEDERAL

SS-G-659 — Graphite, Dry (Lubricating)

MILITARY

MIL-M-7866 — Molybdenum Disulfide, Technical, Lubrication Grade

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

30. MATERIALS

30.1 The molybdenum disulfide specified in this appendix shall conform to MIL-M-7866.

30.2 The graphite specified in this appendix shall conform to SS-G-659, utilizing only that portion passing through a 325 mesh screen.

30.3 The sodium silicate solution specified in the appendix shall have a sodium oxide to silicon dioxide ratio of 1 to 2.9 by weight, shall be approximately 43 percent solids by weight and have a viscosity of 960 centipoises.

30.4 The potassium nitrite specified in this appendix shall conform to certified ACS grade.

The following ratio of ingredients shall be maintained.

MoS ₂ powder	53.3 grams
Graphite powder	18.4 grams
Sodium silicate solution	28.3 grams

Potassium Nitrite	7.3 grams
Water	70.0 grams

40. PREPARATION

40.1 The molybdenum disulfide powder (53.3 grams (g)) and the graphite (18.4) powder shall be mixed in a beaker. Water to which 7.3 g of potassium nitrite has been added shall be added slowly while stirring the mixture thoroughly. Addition of water shall be continued with stirring to insure complete wetting of the powder mixture until a pourable slurry is obtained. The quantity of water shall be approximately 70 g. (Note: An excess of water will result in separation. If separation is observed, the mixture should be discarded and a second preparation using fresh ingredients initiated). The pourable slurry shall be added to the sodium silicate solution (28.3 g) with stirring to obtain a uniform sprayable mixture. The sodium silicate solution should be used within date specified on the container.

50. APPLICATION

50.1 The mixture shall be placed in an 8-ounce capacity spray bottle and the spray bottle attached to a spray gun. The molybdenum disulfide mixture shall be agitated immediately before spraying. The spray pressure shall be approximately 40 psi. The lubricant film shall be allowed to air dry prior to applying subsequent coats.

50.2 The curing schedule shall be as follows:

- a. Air dry for 1/2 hour at room temperature
- b. Heat in oven for 2 hours at 80°C (176°F)
- c. Heat in oven for 2 hours at 150°C (301°F)

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